

Acoustophoretic studies of aqueous suspensions of alumina and 8 mol% yttria stabilised zirconia powders

R. Greenwood*, K. Kendall

Birchall Centre, School of Chemistry and Physics, Keele University, Keele, Staffordshire ST5 5BG, UK

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Abstract

The iso electric points (IEPS) of four alumina powders and three 8 mol% yttria stabilised zirconia powders were investigated using acoustophoresis to measure the zeta potential as a function of pH. All the powders were characterised by X-ray fluorescence spectroscopy for elemental composition and BET nitrogen adsorption for surface area. Initial experiments revealed that the IEPS of the alumina powders were independent of the volume fraction of the alumina suspensions. Further experiments investigated the effect of adjusting the pH with different bases, i.e. NaOH, KOH, and NH₄OH, on the IEPS of the alumina powders. The IEP of an individual powder was independent of the base used, however, the degree of hysteresis varied depending on the polarisability of the cation. The smallest degree of hysteresis was recorded with additions of NaOH. The differences in IEP between the alumina powders were attributed to the different surface chemistries of the powders. For the zirconia powders the IEP of each suspension was identified using HCl and NaOH to adjust the pH. Again the differences in the IEP were attributed to different surface chemistries of the powders. Two of the powders demonstrated an IEP of approximately 6.5; however the Tosoh powder suspension demonstrated considerable hysteresis and an IEP of 9.3. This was due to its natural pH being an approximate pH unit lower than the other two powders. At this lower natural pH, yttria dissolved out of the Tosoh powder, generating yttrium ions in solution. These precipitated onto the powder surface and altered the surface chemistry with time; i.e increased the zeta potential. Atomic absorption experiments on the supernatant confirmed the dissolution of the yttria from the Tosoh powder. © 1999 Elsevier Science Ltd. All rights reserved.

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1. Introduction

In colloidal processing of ceramics it is important to ensure that the ceramic powders are fully dispersed within the medium. Undispersed particles or aggregates cause flaws in the final sintered body which diminish the final mechanical properties.^{1–3} Sub micron sized powder particles aggregate due to the attractive Van der Waals force. However, this can be overcome by the introduction of a repulsive force between the particles. Either by electrostatic repulsion of surface charges or via steric repulsion where adsorbed polymer molecules physically prevent particles coming close enough for the van der Waals attractive force to dominate.

Fortunately ceramic particles are naturally positively or negatively charged depending on the pH conditions.

This amount of charge can be approximated and easily measured via the zeta potential. The zeta potential then is the potential at the shear plane and is generally accepted to be a reasonable measure of the amount of surface charge present. The point at which the zeta potential is zero is termed the iso electric point (IEP). At this pH value the electrostatic repulsive force is zero, hence in a purely electrostatically stabilised system the particles aggregate together. Hence, when designing a stable ceramic suspension it is necessary to have knowledge of the IEP, so that a system has a pH significantly different from that of the IEP. The greater this difference the larger the zeta potentials that can develop to stabilise the system. The kinetic energy of colloidal particles due to Brownian motion is of the order of $3/2kT$, and at 20°C a repulsion barrier corresponding to a zeta potential of about 25 mV is required to minimise coagulation. A larger repulsion barrier or other mechanism for stability is needed to retard agglomeration during pouring and mixing processes which produce a greater

* Corresponding author now at: School of Chemical Engineering, University of Birmingham, Edgbaston, Birmingham B15 2TT, UK. Tel.: +44 (0)121-414-5303; fax: +44 (0)121-414-5324.

kinetic energy.⁴ In some systems, however, acidic conditions may be inappropriate as leaching of impurities^{5–7} or damage to plaster moulds may occur, hence basic conditions are necessary to stabilise the powders. Electrostatic stabilisation is unfortunately not effective at high volume fractions, so traditionally in the ceramics industry where high volume fraction suspensions are utilised, polyelectrolytes are adsorbed onto the powder surface. These probably impart stability via a combination of electrostatic and steric effects.

There are typically two ways of measuring zeta potentials, electrophoresis or acoustophoresis. The former is a well established technique for dilute suspensions. The suspension is subjected to an electric field (strength E) which causes the charged particles to move. The velocity (v) of the particles is then related to the zeta potential via the mobility which is defined as v/E . This mobility is then related to the zeta potential via the Huckel or von Smoluchovski equations depending on the particle size and the ionic strength of the suspension. This is explained further in any standard colloid text book.^{8–11}

Acoustophoresis is a relatively new technique and has the advantage of working at much higher volume fractions than electrophoresis, so that industrially relevant suspensions can be investigated. In this technique an alternating electric field of a known frequency is applied to the suspension which causes the particles to oscillate at the same frequency. The liquid in the shear plane around the particles moves with the particle, but due to differences in permittivity and density the movements are out of phase. This results in a cyclic longitudinal pressure variation in the suspending liquid, i.e. a sound wave. This acoustic wave is detected by the transducers. The amplitude of this acoustic signal and its phase difference is then recorded in the dynamic mobility spectrum, (frequency range 300 kHz to 11.5 MHz). From this spectrum a zeta potential and a particle size can be calculated.^{12–14} Acoustophoresis has been used to study numerous ceramic suspensions,¹⁵ latices and sols,¹³ pigments,¹⁶ clays,¹⁷ coal water suspensions¹⁸ emulsions and semi conducting particles.¹⁹

The purpose of this paper is to study the iso electric points of four different alumina powders and three different 8 mol% yttria stabilised zirconia powders using acoustophoresis. Colloidal processability of the powders could then be compared and optimised.

2. Experimental

2.1. Alumina suspensions

The four alumina powders chosen were Alcoa CT3000SG (Ludwigshafen, Germany), Mandoval AES11C (Mandoval Ltd, Lightwater, Surrey, UK),

SDK160 SG1 and SDK161 SG (both Whitfield and Sons, Stoke on Trent, Staffordshire, UK). The powders were initially characterised for elemental composition by X-ray Fluorescence Optical Emission Spectroscopy (Phillips PW 1606) and for surface area by BET single point nitrogen adsorption (Strohlein Area meter II). Approximately 400 ml of suspension were prepared by mixing the powder with water in one litre plastic bottles at volume fractions of 0.08 and 0.16. The background electrolyte concentration was kept constant at 10 mM KCl (BDH). The suspension was then poured into the sample vessel of the Acoustosizer (Colloidal Dynamics, USA) and allowed to equilibrate at 25°C. The pH was adjusted with HCl (3 M, Timstar, Crewe, Cheshire, UK) and either one of the following three bases: NaOH (3 M, Timstar, Crewe, Cheshire, UK) KOH (2.5 M BDH) and NH₄OH (Ammonia solution specific gravity 0.88, Analytical grade, Fisons). All water used was double distilled. The stirrer speed was kept constant at 300 rpm for the 0.08 volume fraction samples and 500 rpm for the higher volume fraction samples (0.16). The samples were measured within 2–3 hours of preparation and each experiment took approximately 90 min to complete.

Initially, the pH of the suspension was adjusted from the natural pH to one of approximately 10.5, the automatic software was then programmed to decrease the pH to approximately 4 and then increase it back to approximately pH 10. The machine was calibrated in air and with the standard solution at 25°C on a daily basis. The pH meter was calibrated on a weekly basis using three buffer solutions.

2.2. Zirconia suspensions

The three zirconia powders studied were all 8 mol% yttria stabilised zirconia supplied by; MEL Chemicals, (Swinton, Manchester, UK) Tosoh TZ8Y (Whitfield and Sons, Stoke on Trent, UK) and Daiichi HSY8, (Zirconia Sales UK Ltd, Lightwater, Surrey, UK). These were all characterised in the same way as the alumina powders and the suspensions prepared in the same manner. Again the background electrolyte concentration was kept constant at 10 mM KCl. The pH of the suspensions was then adjusted with the HCl and the NaOH only. All suspensions were prepared at volume fractions of 0.08 and the experiments conducted at 25°C. From the natural pH of the suspension the pH was increased to a set point and then decreased to a second set point so that two estimates for the IEP could be obtained. The initial pH and zeta potential were determined by a quick single point experiment. From these values a pH range was chosen for the automatic titration software to cover. For naturally occurring positively charged suspensions, i.e. the Tosoh and Daiichi powders the pH was firstly increased to the set point

then reduced back to another, for the negatively charged MEL powder suspension the pH was initially reduced then increased.

3. Results and discussion

3.1. Effect of volume fraction on the IEP of four alumina powders

The surface areas and impurity levels of the alumina powders have been reported previously.²⁰ Fig. 1 shows a typical plot of zeta potential against pH, for the SDK 161 powder at two volume fractions; 0.08 and 0.16. At low pH values the zeta potentials were large and positive, whilst at high pH levels the zeta potentials were large but negative. There was, however, some degree of hysteresis, with two IEPs being identified as occurring at pHs 7.6 and 8.1, for the 0.08 volume fraction sample and 7.5 and 8.2 for the 0.16 volume fraction suspension. Table 1 records these two IEPs for the four powders at the two volume fractions. The best estimate of the IEP was then taken to be that obtained when decreasing the pH, as the least amount of acid has been added to the system.

From Table 1 the iso electric point of the four alumina powders was noted to be independent of the volume fraction of the suspensions. This confirms that no impurities were dissolving out of the powders and causing the IEP to shift. So further experiments were

Table 1
Effect of volume fraction on the IEP of the four alumina powders

Powder	HCl/KOH volume fraction 0.08	HCl/KOH volume fraction 0.16
Alcoa CT3000SG	8.0–8.9	7.9–8.5
Mandoval AES11C	7.9–9.1	7.8–9.1
SDK160	8.2–9.1	8.1–8.9
SDK 161	7.6–8.1	7.5–8.2

then carried out at the lower volume fraction as this does not waste the powder. If the difference in the two IEPs was taken to be a measure of how strongly flocculated the powder was, then it can be noted that the Mandoval powder flocs appear to be more difficult to break up and that flocs formed by the SDK 161 powder are easier to redisperse. It might be expected that the greater the volume fraction of the suspension then a greater flocculated system would form, which would be more difficult to break down. However, this is not apparent here as the stirrer speed was increased to 500 rpm for the more concentrated samples and the effect is possibly hidden.

3.2. Effect of different bases on the IEP of the four alumina powders

A typical plot of zeta potential against pH is shown in Fig. 2. Three different bases were used to adjust the pH of a suspension prepared from the SDK160 powder at a

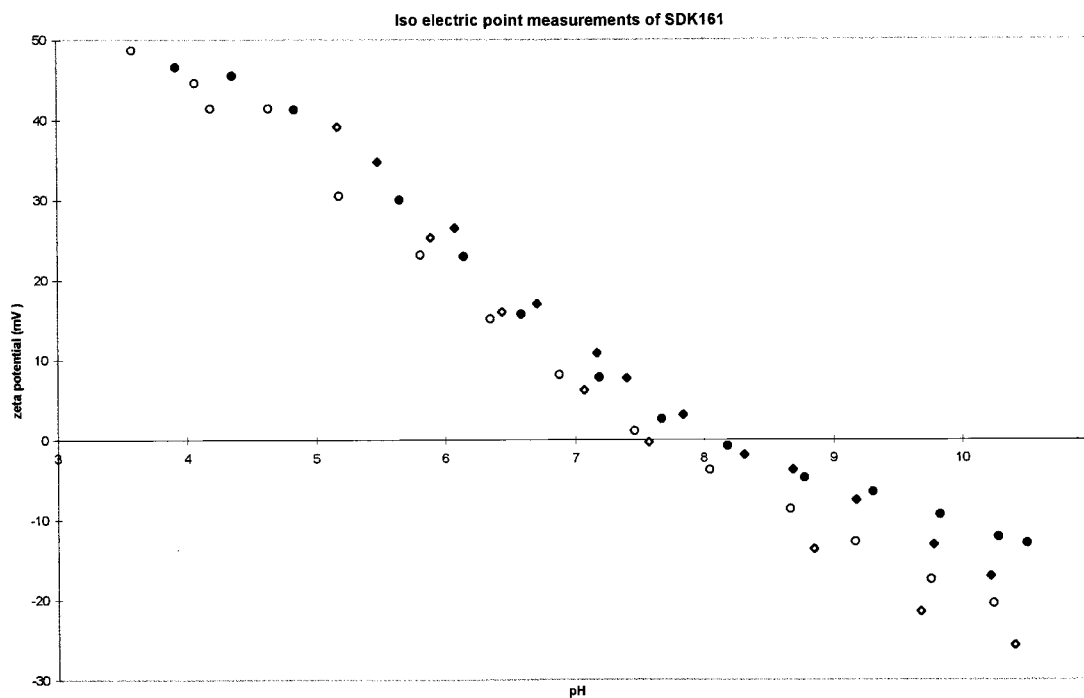


Fig. 1. Effect of volume fraction on the iso electric point of the SDK161 alumina powder. Circles = volume fraction 0.08; diamonds = volume fraction 0.16; filled symbols = increasing pH; empty symbols decreasing pH.

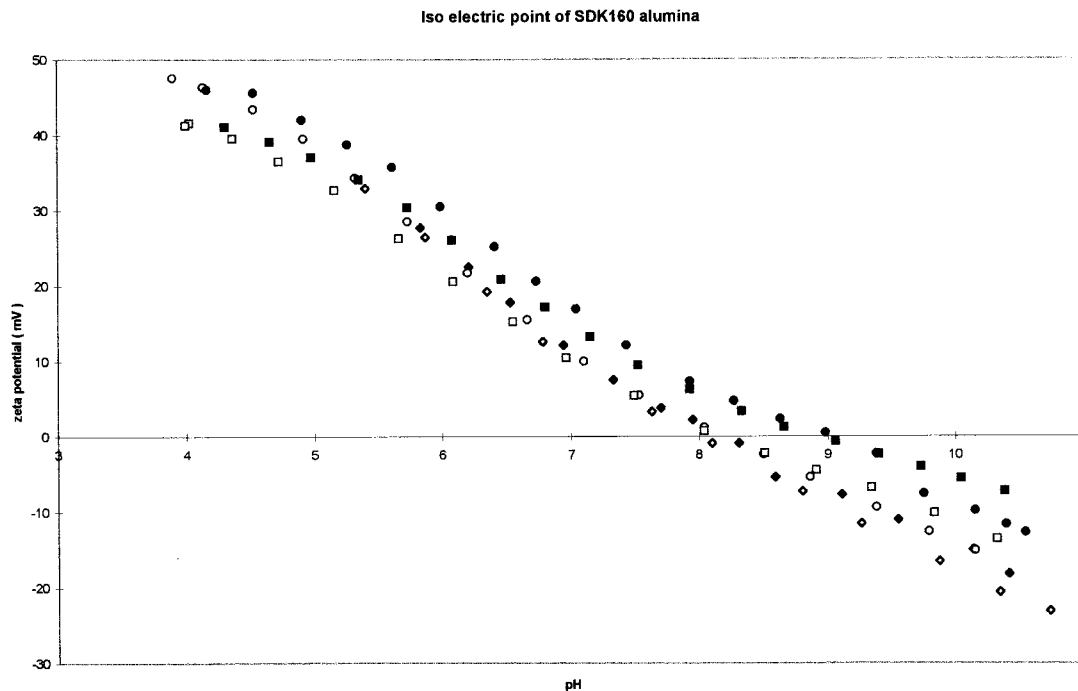


Fig. 2. Effect of different bases on the iso electric point of a suspension prepared from SDK160 powder. Diamonds = pH adjusted with NaOH and HCl; circles = pH adjusted with NH_4OH and HCl; squares = pH adjusted with KOH and HCl; filled symbols increasing pH; empty symbols = decreasing pH.

volume fraction of 0.08. The trends were similar to before in that there was some hysteresis. Again two values for the IEP can be identified, these are listed in Table 2. Again if the lower value for the IEP is taken to be the best estimate, then the results for all three bases are consistent. These agree very well with the results in Table 1. Average mean values for the IEP from all four separate experiments are also listed in Table 2 along with the standard deviations.

From Table 2 it can be noted that the lowest IEP occurs at $\text{pH } 7.55 \pm 0.05$ for the SDK161 powder compared the highest IEP occurring at $\text{pH } 8.12 \pm 0.04$ for the SDK 160 powder. The IEP of the Mandoval and the Alcoa powders were similar, at approximately $\text{pH } 7.9$. The errors here were ± 0.11 and ± 0.05 , respectively. From the XRF spectroscopy data both the SDK 160 and 161 powders have a small mass loss on ignition.²⁰ This could possibly be an organic coating that has been placed onto the particles to tailor a particular surface chemistry and aid dispersion. Bergstrom²¹ reviews the IEPs of several alumina powders and notes that the IEP of alumina varies over the pH range 8–9. So this set of data is in good agreement with the literature. Again if the degree of hysteresis was measured as the difference in the IEP on decreasing the pH compared to that obtained by increasing the pH, then the degree of hysteresis did vary considerably with the different bases used. For suspensions measured using NaOH as the base the degree of hysteresis was small, whereas for

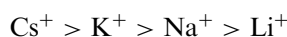
Table 2
Effect of choice of base on the IEP of the four alumina powders^a

Powder	HCl/NaOH	HCl/KOH	HCl/ NH_4OH	Mean pH_{IEP}
Alcoa	7.9–8.3	8.0–8.9	7.9–8.9	7.95 ± 0.05
CT3000SG				
Mandoval	7.9–8.5	7.9–9.1	8.1–8.8	7.92 ± 0.11
AES11C				
SDK160	8.1–8.3	8.2–9.1	8.1–8.8	8.12 ± 0.04
SDK161	7.5–7.7	7.6–8.1	7.6–8.2	7.55 ± 0.05

^a All volume fractions = 0.08.

suspensions measured using KOH and NH_4OH the degree of hysteresis was much larger. But this could be due to the fact that for NaOH the pH range studied was from 5.3 to 10.8 compared to approx. pH 4 to 10.5 for the other two bases.

However this observation is consistent with the lyotropic or Hofmeister series¹⁰. The effectiveness of monovalent cations as coagulants decreases in the following manner



This series is based on the size and polarisability of the ions. Hence in the system where NaOH was used to adjust the pH the particles would be weakly flocculated whereas one in which NH_4OH or KOH was used would be more strongly flocculated, hence would show a

greater degree of hysteresis as the stirrer struggled to break down the more strongly bound flocs. This effect has also been studied by Kosmulski and Rosenholm²² using the Acoustosizer. As before the suspension prepared from the Mandoval powder demonstrated the widest degree of hysteresis and that prepared from the SDK160 powder the smallest, again suggesting stronger flocculation and weaker flocculation, respectively.

3.3. Iso electric points of the three zirconia powders

The XRF spectroscopy results for the three powders are shown in Table 3. All the powders contain 13–14% by weight of yttria as expected, although the exact amount varies. Additionally small amounts of Hafnia were noted in all three samples, but this is not unusual. However, the Daiichi powder had small amounts of P₂O₅ and also a large alumina impurity level (possibly some surface coating). The Tosoh and Daiichi powders also had a small loss of ignition suggesting that perhaps an organic coating was present. Finally a small amount of SiO₂ was detected in all three powders. The surface areas of the powders are listed in Table 4. The three zirconia powders are again quite different, the MEL powder has the smallest surface area suggesting quite a coarse particle size distribution. The Daiichi powder had a slightly higher surface area of 7.4 m²/g, whereas the Tosoh powder had a surface area of 14.3 m²/g, which is consistent with the fact that the powder appears very fine. Previous measurements on a Tosoh 3 mol % yttria stabilised zirconia noted a surface area of 13.2 m²/g.

The zeta potential against pH curves for the three zirconia powders are shown in Fig. 3, and the IEPs are summarised in Table 4. Fig. 3 shows that all the powders had different curves. The suspension prepared from the Daiichi powder had a natural pH value of 6.4 with a

Table 4
Characteristics of the three zirconia powders suspensions

Powder	Initial pH of suspension	Initial zeta potential (mV)	IEP	Surface area (m ² /g)
Daiichi	6.4	+3.4	6.5	7.4
MEL	6.9	−5.5	6.5	2.3
Tosoh	5.6	+23.8	9.3	14.3

small positive charge of +3.4 mV whilst the MEL powder suspension had a slightly higher natural pH of 6.9, but had a small negative charge of −5.5 mV. The Tosoh powder suspension was different again with a natural pH of 5.6 and a large positive zeta potential of +23.8 mV. As before all three powders were highly positively charged at low pH and highly negatively charged at high pH.

Again Bergstrom²¹ reports that the IEP of zirconia varies considerably from pH 5 to 8 and that of pure yttria also varies pH 9–10.6. Reported values for the IEP of 3 mol % yttria stabilised zirconia powders were 5.4–7.7,^{21,24} so it would be expected that 8 mol % yttria stabilised zirconia powders would have a higher IEP, i.e. in the region pH 6–9. It is evident that the IEP of the Daiichi powder occurred at pH 6.5. It is more difficult to report the IEP for the other powders due to the hysteresis, but best estimates would be pH 9.3 for the Tosoh powder and pH 6.5 for the MEL powder. As stated previously it is well known that yttria dissolves out of yttria stabilised zirconia powders under acidic conditions. As two of the suspensions, i.e. those prepared from the MEL and Tosoh powders demonstrated a large degree of hysteresis it is possible there is some dissolution of yttria. It was suspected that as the natural pH of the Tosoh suspension was approximately one pH unit lower than the other two suspensions that the yttria would be dissolving out of this powder in particular. This was then investigated by atomic absorption spectroscopy analysis of the supernatants of all three zirconia powders.

Suspensions of all three powders were prepared (volume fraction 0.08, total volume 43 ml). These were then allowed to stand for 2, 4 and 6 h. After the allotted time the suspension was centrifuged down (5000 rpm for 25 min in a Baird and Tatlock Autobench Centrifuge mark IV) and the supernatant carefully removed with a pipette. The samples were then analysed by Atomic Absorption spectroscopy for seven different impurities. The results of which are shown in Table 5(a)–(c).

The supernatant taken from the Daiichi suspension had very low levels of all the impurities measured, hence no impurities are leaching out of this powder. The supernatant taken from the MEL suspension, however, had detectable quantities of Yttrium, Calcium and Sodium ions. Whilst the Tosoh supernatant had an extremely high Yttrium ion content (170 mg/l) and also high concentrations of Calcium, Sodium and Zirconium

Table 3
XRF results for the three zirconia powders

Oxide	Daiichi (% weight)	Tosoh (% weight)	MEL (% weight)
SiO ₂	0.4	0.05	0.05
TiO ₂	<0.01	<0.01	0.08
Al ₂ O ₃	0.25	0.02	<0.02
Fe ₂ O ₃	<0.01	<0.01	0.01
CaO	<0.01	<0.01	<0.01
MgO	<0.05	<0.05	<0.05
K ₂ O	<0.01	<0.01	<0.01
Na ₂ O	<0.01	<0.01	<0.01
ZrO ₂	84.6	83.9	85.4
HfO ₂	1.72	1.73	1.58
SrO	0.01	0.01	0.02
P ₂ O ₅	0.02	–	–
Y ₂ O ₃	13.2	14.2	12.9
Loss on ignition	0.14	0.10	<0.01

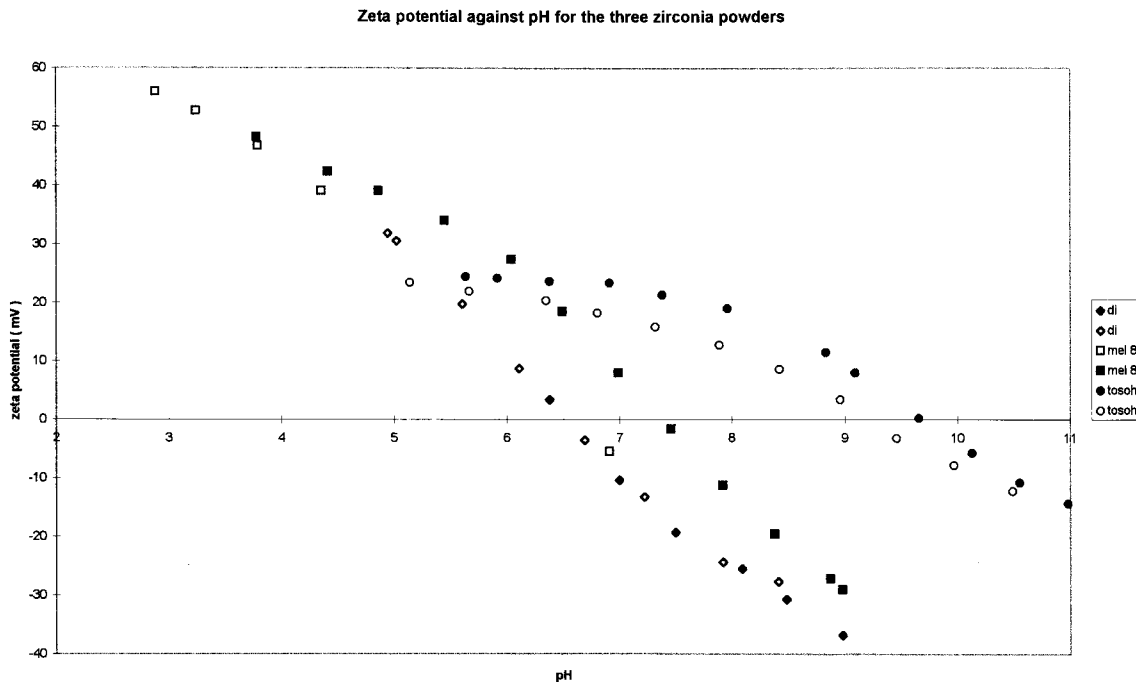


Fig. 3. Iso electric point of three zirconia powders. Diamonds = Daiichi; circles = Tosoh; squares = MEL; filled symbols = increasing pH; empty symbols = decreasing pH.

Table 5
Effect of time on the supernatant of a suspension prepared from the
(a) Daiichi powder; (b) MEL powder; (c) Tosoh powder

Element	2 h (mg/l)	4 h (mg/l)	6 h (mg/l)
(a) Daiichi powder			
Calcium	3	1	1
Aluminium	< 1	< 1	< 1
Yttrium	< 0.2	< 0.2	< 0.2
Sodium	6	6	6
Zirconium	< 0.1	< 0.1	< 0.1
Magnesium	< 1	< 1	< 1
Titanium	< 0.05	< 0.05	< 0.05
(b) MEL powder			
Calcium	13	12	12
Aluminium	< 1	< 1	< 1
Yttrium	15	14	14
Sodium	8	8	8
Zirconium	< 0.1	< 0.1	< 0.1
Magnesium	< 1	< 1	< 1
Titanium	< 0.05	< 0.05	< 0.05
(c) Tosoh powder			
Calcium	34	35	36
Aluminium	< 1	< 1	< 1
Yttrium	170	170	175
Sodium	24	23	24
Zirconium	27	51	40
Magnesium	< 1	< 1	< 1
Titanium	< 0.05	< 0.05	< 0.05

ions. The yttria dissolves out of the Tosoh zirconia powder quickly as after 4 h and 6 h the concentration level does not increase further. Compared to the same

analysis that has been carried out for 3 mol % yttria stabilised zirconias²³ the concentration of yttrium ions is much higher. This would be expected as more yttria (8 mol% compared to 3 mol%) is present to leach out of the zirconia, i.e. a concentration of 170 mg/ml is detected compared to 20 mg/ml for a 3 mol% yttria stabilised powder leaching under the same conditions.

To see if this dissolution of yttria effects the zeta potential of the suspension fresh samples of all three powders were prepared at volume fractions of 0.08 and electrolyte strengths of 10 mM KCl. The zeta potential of the suspension was then recorded every 15 min over a 6 h period, the results of which are shown in Fig. 4. The first data point is taken after 15 min from initial mixing of the powder and water as this was the minimum time taken to reach equilibrium at 25°C. It can be seen that the zeta potential of the suspension prepared from the Tosoh powder increases from +22 to +31 mV over the six hour period. Over the same time period the pH value remained the same, changing slightly from 5.71 to 5.66, whilst the conductivity fell from 3.07 S/cm to 2.38 S/cm over the same time period. The particle size distribution also decreased with time, indicating that the sample was becoming more stable with time. This change in zeta potential of 9 mV is greater than the change expected due to the small change in pH so can be attributed to yttria dissolution forming Y^{3+} and Y^{4+} ions in solution. These ions then precipitate onto the powder surface increasing the overall positive charge.

The results for the Daiichi powder were vastly different to that for the Tosoh powder. The zeta potential

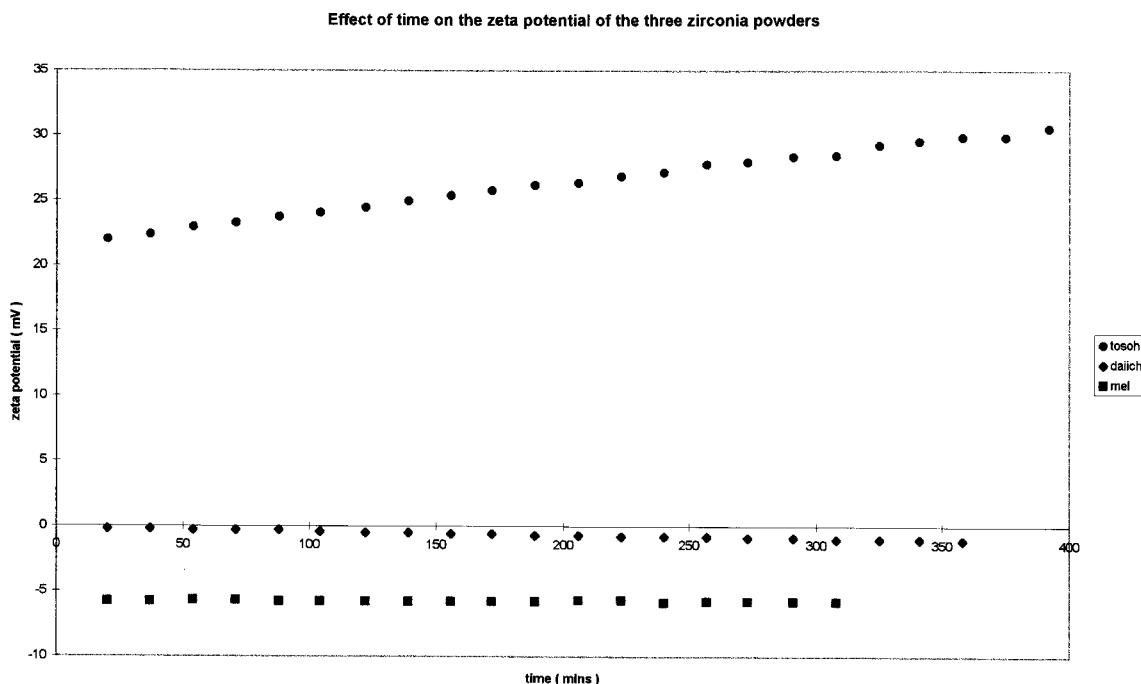


Fig. 4. Effect of time on the zeta potential of the three zirconia suspensions. Diamonds = Daiichi; squares = MEL; circles = Tosoh.

drifted slightly from an initial value of -0.2 to -1.1 mV over the time period studied. However, this is consistent with the corresponding small change in pH from 6.35 to 6.45 over the same time period. The corresponding conductivity change was from 1.40 to 1.52 S/cm. This however does suggest that the IEP measurement from the previous experiment of 6.5 is slightly high, because in this experiment at pH 6.5 the particles are slightly negatively charged, so a better estimate would be 6.3. So it can be concluded that no yttria is dissolving out of this particular powder, which is in agreement with the atomic absorption spectroscopy results on the supernatant.

Similar results to the Daiichi powder suspension were obtained for the MEL suspension, the zeta potential was a constant -5.8 mV and the corresponding pH change was 7.01 to 7.04, whilst the conductivity values decreased slightly from 1.34 to 1.32 S/m. Again this is in agreement with the atomic absorption results which indicated that although there was some yttria present in the supernatant it does not effect the zeta potential of the suspension over the time period studied.

4. Conclusions

The iso electric point of the four alumina powders studied was independent of the volume fraction. The IEP varied from 7.5 to 8.1 depending on the surface chemistry of the powder. Different degrees of hysteresis were also noted for the different powders, the largest

degree of hysteresis occurred with the Mandoval powder and the least with the SDK161 powder. The degree of hysteresis was also dependent on the base used to alter the pH. The smallest degree of hysteresis was obtained using sodium hydroxide to adjust the pH, whilst potassium hydroxide and ammonium hydroxide caused large degrees of hysteresis. This reflects the lyotropic or Hofmeister series which reflects a cations ability to cause flocculation due to its size and polarizability.

In the study of the three 8 mol% yttria stabilised zirconia powders it was noticed that yttria dissolved out of the Tosoh powder at its natural acidic pH. Thus causing the zeta potential of the suspension to increase with time. This presence of yttria ions in solution was confirmed by atomic adsorption spectroscopy experiments on the supernatant. This dissolution was not evident in the other two powders as they had a natural pH near to neutral, hence little or no dissolution occurred.

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